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CUBIC Silicon Carbide (3C SiC): Growth and Properties of Single Crystals and
Polycrystalline Layers PREPARED BY THERMAL DECOMPOSITION OF
METHYLTRICHLOROSILANE IN HYDROGEN

by Stanislav N. GORIN and Ludmila M. IVANOVA

(service lifes of 10000 - 100000 hours) and working temperatures to 700 K; thermal indicators for measuring maximal temperatures in a range of 360 to 1600 K in not easily accessible places at the objects of atomic industry and space technology; high-temperature photodetectors for recording power laser radiation; etc.

The high-purity polycrystalline layers of cubic silicon carbide may be used as protective coatings on items of graphite or commercial (Acheson's) silicon carbide as well as for making various structural elements such as chemically resistant reactors, containers, mirrors, etc., employed in microelectronics and power device-building.

This overview briefly describes the physico-chemical investigations into the process of growing cubic silicon carbide (3C SiC) from the vapor phase by thermal decomposition of methyltrichlorosilane in hydrogen. The method was developed in the Baikov Institute of Metallurgy, Russian Academy of Sciences; it ensures manufacturing single crystals and polycrystalline layers that can be used for the production of various semiconducting devices and passive elements.

2. BRIEF HISTORY

Silicon carbide was first obtained by Berzelius [1] as long ago as in 1824, but the real birth year of SiC is 1892, when it was obtained and investigated almost simultaneously by Moisson [2] and Acheson [3]. The latter, who realized the technical importance of this hard and chemically stable product, has developed a commercial technique [4] that up to now is the main method for the production of silicon carbide. Tens of thousands of tonnes of SiC are manufactured by this method annually, primarily for the needs of abrasive, chemical, and electrotechnical industries, and metallurgy.

The Acheson process yields SiC crystals of the alpha modification. For sure

beta silicon carbide that had no marked conductivity at either room or higher temperatures.

Later (1947 - 1960), Kendall [9 - 11] used the same process in wider composition and temperature ranges. He conducted reactions between silicon tetrachloride and toluene in hydrogen and nitrogen atmospheres and in a partial vacuum; he used mixtures of silicon tetrachloride, benzene, and hydrogen; silicon tetrachloride and methane; silicon tetrachloride + methane + hydrogen; as well as methyltrichlorosilane (MTCS) and diethyldichlorosilane without hydrogen.

Kendall, like Moers, could not obtain crystals of more than 0.3 mm in size because the temperature on the crystal surface decreased with time and, with increasing current, the filament burnt out. All crystals were of cubic modification. However, the small size of crystals prevented direct measurement of electrical properties. The type of the charge carriers was determined from the sign of thermopower [10].

In 1955, Lely [12] reported on the development of a laboratory technique for the preparation of silicon carbide crystals by the sublimation of commercial SiC obtained by the Acheson process. Later [13, 14], Lely studied some properties of pure and doped crystals obtained by his method.

Lely's method permits one to obtain fairly large and perfect SiC crystals of hexagonal modification, but the yield is small and the apparatus is wieldy and power-consuming. For this reason, the attention of researchers was subsequently directed, from the one hand, toward the improvement of this method [15, 16, 24 - 28, 68, 69] and, on the other hand, toward searching out new, cheaper and more efficient, techniques for growing silicon carbide crystals, including 3C SiC. Studies in the development of the Van Arkel process continued [20, 21, 29 - 37]. In particular, attempts were undertaken to obtain SiC crystals inside a tube [31, 32, 35, 38 etc.] rather than on a filament in the gas-suspension state [30]; in an electric arc

By the end of sixties, the attention of researchers was focused on the process of heteroepitaxy of 3C SiC on substrates such as Si, alpha silicon carbide, sapphire, etc., from various starting components (organosilicon compounds, silicon and carbon chlorides, hydrocarbons, etc.) [83 - 88].

In the seventies, the circle of researchers dealing with growing single crystals and polycrystalline films of 3C SiC became broader [75, 81, 89 - 95]; a large group of Japanese authors joined [96 - 98]. New data were accumulating; the properties of 3C SiC were actively studied. As starting components, they used methyltrichlorosilane [96], a mixture of silicon tetrachloride and propane [97]. It was found that 3C SiC films grow better on Si(100) substrates rather than on (111) or (110). Interesting results were obtained upon the deposition of SiC on 6H SiC, using methyltrichlorosilane and toluene [93]. It was found that, in this system, films of both alpha and beta modifications of SiC could be grown. Beta SiC is obtained if a silicon excess in the gas phase exists; the excess of carbon yields alpha SiC or an alpha and beta mixture.

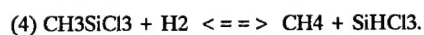
In eighties, Japanese and American researchers suggested the use of a buffer (transition) layer during heteroepitaxial deposition of 3C SiC films on silicon substrates [99 - 102]. This technique was later widely applied in many works; it permits obtaining fairly thick and relatively perfect 3C SiC films (see, e.g., the proceedings of conferences on amorphous and crystalline silicon carbide that were held in 1987 - 1993 [103]).

Because of a very small volume of the overview, we restrict ourselves to this very brief enumeration of the methods that permit producing cubic silicon carbide crystals and consider in more detail the method that was developed in the Baikov Institute of Metallurgy. The principal results of the investigations performed in the Baikov Institute were published in [104 - 126].

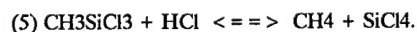
Reactions (1) - (3) do not rule out the possibility of occurring other, side reactions. We studied [112] the composition of the solid, liquid, and gaseous products that formed upon the decomposition of MTCS in hydrogen at $T = 1523 - 1873$ K. The reaction was found to occur with a high yield (60 - 80%) and be accompanied by the formation of the appropriate amount of HCl according to (1). At the same time, reactions leading to the formation of some simple chlorosilanes and hydrocarbons occur.

Chromatography and chemical analysis show that the main liquid products of MTCS decomposition are SiHCl_3 and SiCl_4 . Elevated temperatures favor more complete transformation of MTCS into SiC ; decreasing temperature leads to side reactions that result in the formation of liquid products. In gaseous products of the reaction, small amounts of methane (1.18%), ethane and ethylene (0.31%), and acetylene (0.35%) can be found.

The presence of these compounds can be explained by the occurrence of intermediate and side reactions. For example, upon the interaction of MTCS with hydrogen, trichlorosilane and methane can form by reaction



The interaction of MTCS with HCl that evolves by reaction (1), silicon tetrachloride and methane can form by reaction



The calculation of the changes in the Gibbs free energies for reactions (4) and (5) shows that the reactions can occur in a wide temperature range (300 - 1000 K). The probability of their occurrence decreases with temperature, but they are still possible at above 1000 K, for the Gibbs free energy still remains negative.

We see, thus, that although hydrogen does not participate in the total

(3) the diffusion range (above 1873 K). The activation energy decreases to about 12 ccal/mol at $T = 1873$ K.

Thus, in the range of relatively low temperatures (up to 1723 K), where polycrystalline layers of 3C SiC form, the rate of crystallization is reaction-controlled; at higher temperatures (above 1873 K), where separate crystals form, the limiting stage of SiC growth is diffusion, which controls the rate of the supply of the reacting agents to the surface of growing crystals and the rate of the removal of the reaction products.

4. LABORATORY APPARATUS FOR GROWING CUBIC SILICON CARBIDE SINGLE CRYSTALS AND POLYCRYSTALLINE FILMS

The laboratory apparatus used for growing pure and doped 3C SiC by thermal decomposition of methyltrichlorosilane (MTCS) consists of an all-glass, air- and vacuum-tight system fabricated primarily from hard borosilicate glass, quartz, and Teflon. We avoided using metallic parts because MTCS is a very reactive substance. The dismountable parts of the apparatus, such as the vessels for MTCS and liquid doping compounds (evaporators), are connected to the setup through ground-glass joints or rubber O-rings ensuring the air-tightness of the apparatus. The system permits the introduction of flow-metered streams of hydrogen, saturated with the vapors of MTCS and doping compounds, into the reaction chamber. All flowmeters are calibrated in situ. The apparatus has a modular construction, which ensures rapidly testing and disassembling the setup for cleaning. Each of the moduli can be evacuated and washed by hydrogen independently of the other parts.

The high temperatures developed in the reaction zone (1700 - 1900°C) and the presence of gaseous hydrogen chloride that forms by reaction (1) impose severe restrictions on the chemical stability and purity of the parts located in the reaction chamber. Quartz, graphite, and Teflon serve as the main

merged into Dewar vessels with liquid nitrogen; this permits one to simultaneously remove all condensable and adsorbable gaseous impurities.

The method is based on the high absorption capacity of silica gel and activated carbon at low temperatures; thus, it is known that at -195°C , one gram of activated carbon can absorb 155 cm³ of nitrogen, 230 cm³ of oxygen, 133 cm³ of hydrogen. The content of oxygen and nitrogen in hydrogen purified by this method is known to be less than 10-8 vol. %.

When purifying hydrogen used for growing silicon carbide, special attention should be given to the removal of nitrogen because this impurity is the main shallow donor impurity in SiC. According to literature data, even the best of activated carbons adsorbs nitrogen less effectively than silica gel. For this reason, we used a system of three sequentially connected columns to purify hydrogen. The first column is filled with silica gel and is used for preliminarily drying and purifying commercial hydrogen supplied from a balloon. The second column is filled with activated carbon; we cannot manage without this column because silica gel adsorbs oxygen much worse than activated carbon does, and we know that oxygen, like nitrogen, is electrically active in 3C SiC [123]. The third column is again filled with silica gel; this is intended for improving the purification of hydrogen from nitrogen and also serves as a mechanical filter for trapping carbon dust that can be carried-over with hydrogen from the column with activated carbon.

Before the silica gel and activated carbon are placed into the columns, they must be carefully cleaned in chemically pure or high pure hydrochloric acid (1 : 1), then must be washed in doubly distilled water until any traces of chlorine ions vanish, and dried first in a hydrogen flow and finally in a vacuum. The columns must be "trained" (evacuated to a residual vacuum of an order of 10-2 mmHg) for 0.5 - 1.0 h prior to each run without heating and after each 5 - 10 runs, with heating to $90 - 120^{\circ}\text{C}$ for 3 to

chamber through a water-cooled ground-glass joint (to facilitate cleaning it).

The as-assembled (soldered) apparatus is evacuated with a roughing-down pump until the apparent leakage (i.e., the fall of vacuum after pumping is stopped, caused by the presence of water vapors and other volatile impurities in the setup) disappears. The adequately purified setup is capable of keeping vacuum not worse than 0.1 mmHg for a few weeks if the initial vacuum was 0.01 - 0.001 mmHg.

The growth process may be initiated after the starting components (methyltrichlorosilane and the doping compounds if necessary) are put into the evaporators and the whole apparatus is washed with purified hydrogen. The process consists of passing a vapor-gas mixture of a specified composition at a constant flow rate through the reaction chamber past the filament heated to a specified temperature.

The growth of crystals in a flow system is controlled by many parameters such as the filament temperature; reaction mixture composition; flow rate of the gaseous mixture (and the linear velocity of its motion in the chamber); temperature gradient; the shape of the heated surface; conditions for crystal nucleation; and some other factors such as the effects of impurities, thermal and concentration shocks, etc.

In order to determine the optimal conditions for growth single crystals and polycrystalline films, large systematic work was done.

Using temperatures below 1650°C results in the deposition of numerous small crystals grown together or, at temperatures 1250 - 1450°C, of a continuous dense polycrystalline film. At 1650 - 1750°C, separate euhedral crystals grow, which, frequently, have the form of regular hexagonal well-faceted platelets 3 - 5 mm in length and 1 - 1.5 mm in thickness.

After the growth run is stopped, the hydrogen flow rate is increased to 200 - 300 liter/h, the filament is switched-off, and the system is blown out with hydrogen for at least 0.5 h in order to carefully remove the undecomposed MTCS and the products of its decomposition.

5. GROWTH MORPHOLOGY OF BETA SILICON CARBIDE SINGLE CRYSTALS PRODUCED BY THERMAL DECOMPOSITION OF METHYLTRICHLOROSILANE

At temperatures of about 1650 - 1750°C, the deposit consists, as was mentioned above, of separate faceted crystals. In the general case, crystals have various shapes (which frequently does not exhibit visible symmetry) and various degrees of perfection, which depends both on the growth conditions and the type of the crystal. However, it was found that the majority of crystals obtained under any conditions could be classified to three main types, which were called "platelets", "prisms", and "skeletal crystals". Crystals of the first type are flattened in one of the $\langle 111 \rangle$ directions and frequently have a characteristic form of regular hexagonal platelets with one most developed mirror face. Crystals of the second type are extended along a direction $\langle 110 \rangle$ and have from 6 to 10 faces of this crystallographic zone (pseudoprism faces). Crystals of the third type have a few reentrant dihedral angles that give the crystal a characteristic skeletal appearance.

The facets were identified by X-ray diffraction, etching, and goniometry (performed on a Goldschmidt two-circle optical goniometer with an accuracy of 1 min of arc). An investigation of the morphology and sectorial structure (i.e., the structure of growth cones or pyramids caused by impurity segregation) of pure and doped crystals of 3C SiC in various sections showed that

(1) the growth direction of the majority of crystals is $\langle 110 \rangle$;

(2) the following faces are formed on most crystals: $\{111\}$ (positive

strong birefringence when being inspected in polarized light. It was shown that even most perfect crystals contain a characteristic structure (that was called twin structure) in the form of a few MTL passing through the crystal vertex looking in the growth direction.

Skeletal crystals is a special case: they contain, in addition to coherent twin boundaries on $\{111\}$ planes, twin boundaries of higher order (symmetric third order tilt boundaries of the $(110)[110]$ type with a tilt angle of 31.035° , twin plane $\{552\}$). These boundaries appear as a result of multiple twinning on nonparallel $\{111\}$ planes. From four possible types of the mutual arrangement, only one type occurs in all crystals without exception. Etching and goniometry indicate that the boundaries are of the type $\text{Si}\{552\} - \text{Si}\{552\}$, i.e., they contain dislocations with "wrong" bonds of the A - A type (alpha dislocations). This is a direct consequence of the crystallographic polarity of the sphalerite structure. Another related effect of the polarity is the absence of multiple twins with the apparent fivefold symmetry that are quite typical for diamonds. Multiple twins in 3C SiC always involve the above-described $\{552\}$ -type boundaries.

A comparison of the faces appearing on real crystals with those characteristic of the ideal form shows that MTL can locally increase growth rate of 3C SiC crystals. Measurements of real growth rates of various faces by studying sectorial structure of crystals revealed that

(i) MTLs have their own growth rates V_{MTL} ; when an MTL intersects a face whose growth rate is smaller than $V_0 = V_{\text{MTL}} \cos \alpha$, where α is the smaller angle between the MTL and the face, the face transforms; it is replaced by two induced vicinal faces whose growth rates are related to V_0 as $V = V_0 \cos \beta$, where β is the angle between the induced and vicinal faces. The site where the MTL emerges onto the surface of the face coincides with the edge between the induced faces (which form a ridge). For the $\text{Si}\{111\}$ faces, whose growth rate is about $V_{\text{MTL}}/3$, the tilt of the vicinal faces

$\{111\} \pm \alpha$ relative to $\text{Si}\{111\}$ is $0.5 - 1.0^\circ$. For the $\text{C}\{111\}$ faces, whose

faces. Microtwin lamellae disclose this shell, and the habit and faceting of all types of 3C SiC crystals are unambiguously controlled by their twin structure, i.e., by the thickness of the MTLs present and their type (i.e., by whether the number of twin planes is even or odd).

6. EFFECTS OF CRYSTALLOGRAPHIC POLARITY: IMPURITY SEGREGATION AND ETCHING

Pure beta silicon carbide single crystals are transparent and amber yellow. Some impurities make them dark (black or green). This permits one to easily follow their behavior in crystals. It was found by visual inspection that Al, B, and N are distributed in crystals inhomogeneously: the crystals have "sectorial" structure caused by preferential absorption of different impurities by different faces. The study of the sectorial distribution of impurities was performed in sections cut along growth pyramids of crystal faces (which are also called growth loci or growth cones) prepared in such a way that the section pass through the initial point of growth (the "root" of the crystal by which the crystal is attached to the graphite filament). The distribution of nitrogen and boron was studied in detail. Because both impurities are electrically active in SiC, their distribution found visually was confirmed by electrical measurements.

It was found that nitrogen is "absorbed" preferentially by $C\{hhl\}$ and $C\{hkk\}$ faces and much weaker, by $Si\{111\}$, $Si\{211\}$, and $\{100\}$ faces, whereas boron is absorbed most actively by $Si\{111\}$ faces, somewhat weaker by $Si\{211\}$ faces, and much weaker, by $C\{hhl\}$, $C\{hkk\}$, and $\{100\}$ faces, i.e., preferential absorption of nitrogen is observed in growth pyramids of "carbon" faces (positive hkl forms) and it is absent in "silicon" faces (negative hkl forms); for boron, the picture is opposite: this impurity is preferentially absorbed by negative hkl forms. Cube faces $\{100\}$, which are nonpolar in the sphalerite structure, exhibit no preferential absorption for either nitrogen or boron. Aluminum is distributed similar to boron.

triangular etching figures at the faces of negative hkl forms (carbon faces), whereas such figures are absent at the faces of positive hkl forms (silicon faces); moreover, the etching figures are depressions on silicon faces and they are hills on carbon faces. Etching pits on cube faces are lens-shaped, with lens axes aligned in only one of the two possible $\langle 110 \rangle$ directions; etching pits at the opposite $\{100\}$ faces are mutually perpendicular. In some etchants (PbO. chlorine), the etching rates of carbon faces are many times greater than those of silicon faces. Upon oxidation in wet oxygen (at 1200°C), the oxide film on carbon faces is approximately twice as thick as that on silicon faces.

7. POLYCRYSTALLINE LAYERS OF CUBIC SILICON CARBIDE: PREPARATION AND PROPERTIES

The yield, deposition rate, and structure of polycrystalline layers of 3C SiC (PCL) that are obtained by reaction (1) at low temperatures depend on a number of parameters, the most important of which are (a) the temperature at which the deposition is performed (T, °C); (b) the concentration of the starting component in the gas phase (C, g/liter); (c) the flow rate of hydrogen in the reaction chamber (V, liter/h); and the area of the deposition surface (S, cm²). When studying the growth of PCL in our work, these parameters were varied in the following limits: the deposition temperature, 1250 - 1600°C; the MTCS concentration in hydrogen, 0.2 - 1.7 g/liter; and the gas flow rate, 3 - 30 liter/h.

Our investigations [105, 113] indicate the following conditions to be optimum for the production of pure dense polycrystalline layers of 3C SiC: the deposition temperature, 1400 - 1500°C; the concentration of MTCS in hydrogen, 0.5 - 1.5 g/liter; and the flow rate of the gaseous mixture, 6 - 10 liter/h.

Under these conditions, the 3C SiC PCL have a smooth surface and fine microstructure: the yield of SiC varies from 65 to 70%. The duration of the

reflectance in the range of 10 to 14 microns: $R = 90\%$.

Measurements of the electrical properties show that pure 3C SiC PCLs have electronic conductivity; their resistivity varies from 0.1 to 1000 Ohm cm; the charge carrier concentration is $n = 1.4 \times 10^{16} - 5 \times 10^{17} \text{ cm}^{-3}$.

Pure polycrystalline beta silicon carbide has high thermal conductivity. The maximum value is 5 W/(K cm) at 110 K; with increasing temperature, thermal conductivity decreases, but still remains sufficiently high: 2.5 W/(K cm) at room temperature.

The thermopower of pure polycrystalline beta silicon carbide with a charge carrier concentration of $n = 5 \times 10^{17} - 1.8 \times 10^{16} \text{ cm}^{-3}$ ranges from -500 to -600 microvolt/K.

8. DOPING POLYCRYSTALLINE CUBIC SILICON CARBIDE

Doping polycrystalline beta silicon carbide with nitrogen and boron was performed directly during growth by thermal decomposition of methyltrichlorosilane.

The most suitable compounds for doping with nitrogen are nitriles, which are the derivatives of aliphatic and aromatic acids. They only contain carbon, hydrogen, and nitrogen, i.e., there is no danger of contamination by foreign impurities when using nitriles. The nitriles are stable compounds that can be purified by distillation or rectification in air under an atmospheric pressure.

From the variety of nitriles available, we selected acetonitrile as the simplest one [114, 118]. In order to introduce it into the reaction chamber, a separate evaporator was used, which permitted us to easily vary its concentration in the gas phase

(c) all samples grown at boron concentrations lower than 1.5 mg/liter retained electronic conductivity. Distinct inversion of the conductivity type from n to p type occurs at a boron concentration of 2.5 mg/liter. The samples had in this case a resistivity of 2600 Ohm cm. According to activation analysis, the maximum content of boron in beta SiC is about $4 \times 10^{19} \text{ cm}^{-3}$;

(d) the introduction of excess silicon (in the form of silicon tetrachloride or trichlorosilane) into the gas phase strongly affects the process of doping beta SiC by both donor and acceptor impurities, decreasing the degree of doping by an order of magnitude or even greater.

Doping with boron substantially improves photoelectric properties of polycrystalline samples of 3C SiC [125]: it increases the impurity conductivity in comparison with that of pure beta SiC; it increases the exponent in the expression for the lux-ampere characteristics for both the intrinsic and extrinsic conductivities; the temperature dependence of the photocurrent becomes activative (the photoresponse increases with increasing temperature); the activation energy increases with increasing concentration of boron. The photoresponse times are much shorter in these samples (of about few seconds).

The photoelectric properties of polycrystalline cubic silicon carbide may be of interest for the production of photodetectors that can operate under extreme conditions such as high temperatures, high pumping levels, reactive media, severe irradiation, etc.

9. SOME PARAMETERS AND APPLICATIONS OF PURE 3C SiC SINGLE CRYSTALS

Single crystals of pure cubic silicon carbide prepared by thermal decomposition of methyltrichlorosilane in hydrogen have the following

= working temperature: -60 to +80°C.

(2) High-temperature photodetector.

It can be used for detecting high-power laser radiation; operating at elevated temperatures; precision detecting of short-wavelength light signals (up to ultraviolet range). Its main parameters are as follows:

= spectral range: 280 - 650 nm;

= spectral maximum: 330 - 480 nm;

= working temperature: 20 - 350°C;

= photosensitivity: 10 mA W⁻¹ in valve regime; 1000 mA W⁻¹ in photoinjection regime; 50 mA W⁻¹ in photodiode regime;

= response speed: to 5×10^{-7} s;

= service life: > 105 h;

= maximal irradiation power: 40 W (stationary);

100 W (pulsed);

=

(3) Gamma detector.

A high-temperature detector of high-power radiation flows; intended for detecting gamma radiation (> 10⁸ rad) in temperature range from -200 to +400°C. It has a highly linear dynamic characteristic; no degradation under high irradiation doses.

(4) Violet emitter.

May be used as a standard light source for calibration and stabilization of photometric tracts in opto-electronic systems.



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s in crystals (sectorial structure).

On the basis of the platelet-type crystals of 3C SiC grown in the Baikov Institute of Metallurgy, a number of devices were developed in the Kiev Polytechnical Institute, such as light-emitting diodes of high brightness, high-temperature photodetectors, gamma-radiation detectors, violet emitters, etc.

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